

SURFACE EFFECTS ON THE PLASTIC DEFORMATION OF ALUMINIUM SINGLE CRYSTALS

BY

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DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY
KANPUR
FEB., 1973

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SURFACE EFFECTS ON THE
PLASTIC DEFORMATION OF ALUMINIUM SINGLE CRYSTALS

A Thesis Submitted
In Partial Fulfillment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

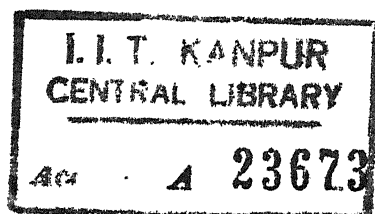
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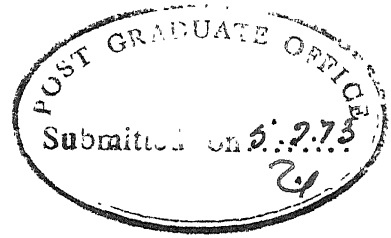


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CERTIFICATE



It is to certify that this work " Surface Effects on the Plastic Deformation of Aluminium Single Crystals " has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

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ACKNOWLEDGEMENT

I wish to record here my deep sense of indebtedness to Dr. G.S.Murty for his invaluable help throughout the period of this work. His keen and critical approach to research had inspired me during the various phases of this work.

I also wish to record my thankfulness to Mr. Krishnamurthy of Crystal growth Lab. for keeping at my disposal his excellent engineering skill during the period of crystal growth.

I also thank my friend Mr. T.K.Sharma for his comradely help throughout the period of this work.

I also thank Mr. C.V.Raju of Chemical Engineering for his patient typing of the manuscript of the thesis.

Above all I cherish the invaluable help of my comrades and friends without whose help this work would have been impossible.

T.R. Ramasami

Author

The author also wishes to thank Mr. RAHMAN of Materials Testing Laboratory for his ready help with the INSTRON and all testing equipment and Mr. D.C. Verma of Civil Engg. for his excellent drawings.

T.R. Ramasami

ABSTRACT

The importance of surfaces in plastic deformation of metals is well established. In spite of several studies made regarding surface effects the exact dislocation mechanisms that lead to such effects are not yet clearly understood. For metals with clean surfaces as well as in metals with surface films the results obtained in the past are contradictory. An example of such a contradiction in metals with clean surfaces is the evidence for the formation of hard as well as soft surface layers due to plastic deformation. In the present investigation the surface effect on plastic deformation of Al single crystals (99.9% pure) were studied in order to clarify the operative dislocation mechanism. Al single crystals with preferred orientation as well as random orientation were deformed in tension with and without anodized oxide layer~~x~~ on the surface. Effects of intermittent surface dissolution and/or intermittent anodizing were also investigated. Stress relaxation tests in air as well as when surface layers were being dissolved were also carried out. ✓

It was found that surface oxide layer always leads to strengthening. The effect of surface dissolution on flow stress was found to depend on the strain level . At strains below about 0.06 surface dissolution led to a rise in flow stress whereas surface dissolution above 0.06 strain resulted in a decrease in flow stress. On oriented single crystals when the E sides through which edge dislocations emerge predominantly were anodized a marked increase in flow stress

was observed, whereas when the S sides through which predominantly screw dislocations emerge were anodized no change was observed. Similarly when E sides were dissolved at higher strains the flow stress dropped whereas dissolution of S sides produced no change in flow stress. At lower strains (below about 0.06) dissolution of E sides and S sides resulted in a rise in flow stress.

The strengthening effect of the oxide film seems to be due to the film acting as a barrier to dislocation egress. This conclusion is supported by the results of stress relaxation test and dissolution of surface layers at higher strains. The rise in flow stress observed at lower strains on surface dissolution is indicative of soft surface layers in contrast to the other results. The results of tests on edge oriented crystals establish the dominant role played by ^{edge} dislocations in strain hardening. Thus ^{the} strain hardening rate is more sensitive to the glide distance of edge dislocations.

CONTENTS

	Page
Acknowledgement	ii
Abstract	iii
CHAPTER	
I INTRODUCTION AND LITERATURE SURVEY	1
II EXPERIMENTAL PROCEDURE	24
(a) Crystal Growth	
(b) Crystal Cutting	
(c) Preparation of Tensile Specimens	
(d) Mechanical Testing	
III RESULTS AND DISCUSSION	38
(a) Crystals with Preferred Orientation	
(b) Randomly Oriented Specimens	
IV CONCLUSIONS	48
V BIBLIOGRAPHY	49

LIST OF PLATES

No.		FIGURE	PAGE
1	Schematic illustration of (a) debris layer model (b) surface source model (c) near surface source model	1	52
2	Effect of surface removal on yield stress	2	53
3	Relative effects of incremental polishing and impacting with silicon carbide particles on stress/strain curve	3	53
4	Schematic illustration of the crystal orientation used by Kitajima in etch-pitting tests	4	53
5	Variation of dislocation density as a function of distance from the (111) surface in the crystals	5	53
6	Effect of active surface dissolution on the stress/ strain curve of nickel monocrystals	6	54
7	Illustration of E-sides and S-sides of Al crystals in Nakada & Chalmers work	7	54
8	Flow stress distribution as a function of distance from surface in strained crystals	8	54
9	Effect of Ni-Cr film on stress/strain curve of similarly oriented copper monocrystals	9	55
10	Effect of passive film on the flow behaviour of nickel monocrystals	10	55
11	Effect of silver plating and an alloyed surface layer on stress/strain curves of copper monocrystals	11	55
12	Influence of the character of emergent dislocations on surface hardening of Al monocrystals	12	55

NO

FIGURE PAGE

13	Transmission electron micrograph showing dislocation network due to mismatch at the interface of a platinum deposit on gold	13	56
14	Stress/strain behaviour of unplated and nickel plated copper monocrystals	14	56
15	Brass grips	17a	59
16	Grip assembly	17b	59
17	Tensile test specimens of Al monocrystals	18a	59
18	Spherical single crystal seed	18b	59

LIST OF FIGURES

NO		FIGURE	PAGE
1	Cutout diagram showing parts of crystal growth assembly	15	57
2	3-Channel mould (Graphite)	16	58
3	Steriorgraphic triangle showing orientation of single crystals	19	60
4	Types of single slip orientation used	20	61
5	Effect of anodizing S-sides of the Al single crystal	21	62
6	Effect of anodizing the E-sides of the Al single crystal	22	63
7	Effect of dissolution of E-sides on the stress/strain curve	23	64
8	Effect of dissolution of S-sides on the stress/strain curve	24	65
9	Effect of anodizing and dissolution of E-sides on the stress/strain curve	25	66
10	Effect of anodizing and dissolution of S-sides on the stress/strain curve	26	67
11	Effect of surface oxide layer on stress/strain curve of Al single crystal	27	68
12	Effect of surface oxide layer on stress/strain curve of Al single crystal	28	69
13	Effect of surface removal at later stages of deformation (Above 0.06 strain)	29	70

NO		FIGURE	PAGE
14	Effect of anodizing and surface dissolution at later stages of deformation	30	71
15	Effect of surface removal in chemical reagents(3%NaOH)	31	72
16	Effect of surface removal and anodizing on the early stages of deformation (Below 0.06 strain)	32	74
17	Effect of surface dissolution on the early stages of deformation (Below 0.06 strain)	33	75
18	Study of sub surfacedamage if any due to anodizing and its effect on stress/strain curve	34	76
19	Effect of subsequent anodizing on the stress/ strain curve	35	77
20	Stress relaxation test in air and 3% NaOH Load vsElongation diagram	36	78

INTRODUCTION AND LITERATURE SURVEY

Many of the solid state phenomena thought of to be properties of bulk material really involve the surface or near surface regions of the materials. Especially the mechanical and corrosion properties of materials are influenced by surface and environmental conditions. We have as the simplest example the case hardening in steels. There are experimental evidences that a thin alloyed layer at the surface makes the bulk behave as though whole of it is alloyed. A thin layer of oxide on aluminium considerably improves its mechanical and corrosion properties. There are numerous examples to illustrate the surface properties.

Though a much more clear understanding of the atomistic and electronic nature of surfaces is necessary to explain such complicated phenomena like Rebinder effect, liquid metal embrittlement, electro capillary effect a simple mechanistic approach is deemed to be sufficient to explain simpler phenomena concerning the way surface nature effects plastic deformation in metals and consequently the many important mechanical properties of metals.

Experiments involving tension, creep, fatigue, torsion, have been conducted on single as well as polycrystals of various metals with their surface natures modified by different treatments. Thus specimens have been studied with surface oxide layers, surface alloy layers, surface films both electro deposited and evaporated and

surface dissolution both continuous and intermittent. Also studies involving plastic deformation in changed environmental conditions have been carried out such as immersion in surface active agents, in vacuum, and in different gaseous atmospheres at controlled pressure

Apart from their interesting practical applications, studies involving surfaces have their importance in the theoretical considerations of plastic deformation. There is considerable dispute regarding the various aspects involved in plastic deformation of metals. The nature and degree to which surfaces influence the basic mechanisms involved in plastic deformations has been the topic of intensive research. The following is a comprehensive survey of literature of works regarding mainly the surface dissolution and surface film effects. The mechanical behaviour of crystalline materials is largely determined by the generation, motion, and interaction of dislocations. Surfaces can act as regions where dislocations are readily generated or their motion impeded. These effects are modified in the presence of surface films of different nature or when the surface is exposed to environments from which surface active species may be adsorbed. Consequently in vacuum in the absence of species it can be expected that the properties will considerably change.

The study of the effect of surface condition on the mechanical properties of metal can be done simply by altering the conditions of the surface or its environment so as to affect the generation, motion

and interaction of dislocations . One can readily see that covering the surface with films of different kind or removal of such films, removal of surface itself either intermittently or continuously are processes by which surface nature can be considerably altered. On the other hand testing in vacuum or at known pressures with controlled atmosphere having known species of gases and testing in electrolytes or solutions containing surface active agents are methods where the environmental conditions are altered.

In general the results of such experiments are explained on the basis of three theories of dislocation generation. The essential differences between these models is illustrated in Fig. 1. These three mechanistic models differ fundamentally in terms of the type of location of dislocations sources considered to be primarily involved in the deformation process.

That utilized by Kramer and coworkers shown in Fig. 1-a (1 - 9 Ref.) assumes that dislocations are generated at internal sources and traverse out. As they approach the surface a fraction of them get trapped near the surface forming a zone of high dislocation density. This Kramer calls the "debris layer". Now this "debris layer" acts as a barrier for further dislocation egress. It is difficult to appreciate how the dislocations pile up at clean surfaces. Shchukin ^{Ref} observes that a force tending to pull an edge dislocation out of the surface is resisted by ^{the} work of forming new surface area whereas

screw dislocation can move out. This ~~force~~ ~~resisting~~ ~~egress~~ ~~is~~ ~~always~~ greater than image force according to Shchukin. Then it is possible for the nascent surface to support a pile up of edge dislocations.

Others assume, Fig. 1 - b, that dislocations are first generated by the surface sources. Since a source pinned in the crystal having its other end terminating at the surface unpinned acts as a Frank-Read source twice its actual length with an image equal to its real length acting as the other half. Since the stress required to activate a source is ^{inversely} ~~directly~~ proportional to its unpinned length such a surface source will be activated at half the stress value of a source in the bulk with both of its ends pinned. So a surface source in all probabilities is the one which is activated in the initial stages and half loops traverse inwards away from the surface.

A third approach is that of Kuhlmann-Wilsdorf(14) and has been discussed by Latanision and Staehle (15, 16). They consider near surface sources to be the most likely generators of dislocations in the early stages of deformation. Such sources are thought to be of the Frank-Read type and are supposed to reside within few microns from the surface. Calculations have shown (17) that such sources illustrated in Fig. 1 -c, operate easily and should be most probable dislocation generators in the early stages of deformation. They generate loops, portions of which escape out of the crystal easily because of their proximity to the surface and the other portions move into the

interior. However they propose that for such sources surface can still act as a barrier though as in the first model how this can occur is not clear.

The various phenomena observed involving surfaces can be seen in the light of these three models.

SURFACE-REMOVAL: The effect of surface removal has been studied extensively by Kramer and coworkers (1 - 9). (1) They report that when aluminium mono crystals were pulled in tension during dissolution in an electrolytic cell the extent of stages I and II of work hardening increased and the stress at which stage III began decreased and work hardening coefficient in all 3 stages decreased (1). The influence of surface removal on these parameters was even more marked when the removal rate was increased upto a maximum of $0.2 \mu / \text{sec}$. Increasing the strain rate at a constant rate of metal removal reduced the effectiveness of this treatment (1). (2) Critical resolved shear stress was not affected by changes in the metal removal rate. (3) The initial yield stress and work hardening behaviour of aluminium crystal deformed into stage I could be recovered by surface removal, Fig. 2. Kramer reports this behaviour in gold mono crystals as well.

Surface removal effects and other effects are sought to be explained by Kramer on the basis of the existence of a preferentially work hardened surface layer where the dislocation density is higher than that in the interior of the crystal. This is the "debris layer"

which serves as a barrier for dislocations against which they pile up to exert a back stress. The net stress τ^* acting on a mobile dislocation can be represented as

$$\tau^* = \tau_p - \tau_i - \tau_s$$

where τ_s is the back stress associated with debris layer

τ_i is the stress associated with the presence of internal obstacles,

τ_p is the applied plastic stress. $\tau_p = \tau_a - \tau_0$

where τ_a applied shear stress, τ_0 is the CRSS.

τ_s has been determined by ~~deforming~~ specimens to a given strain unloading and removing a predetermined amount of material from the surface electrolytically. The difference between the final flow stress and initial flow stress upon reloading is taken as a measure of the decrease in τ_s due to removal of debris layer in whole or in part. Such experiments indicate that debris layer can extend to a depth of 60-100 microns (μ) and that τ_s increases linearly with strain in stages II and III. Kramer concludes that work hardening in stage I is confined primarily to surface layers which implies that transition to stage II will occur at lower strains for smaller crystals. But in general it appears, reverse is true (18,19). Fabiniak and Kuhlmann-Wilsdorf (20) have also reported recovery of the initial yield stress by removing 50 - 100 μ from the surface of lightly deformed aluminium mono crystals. In contrast however Nakada & Chalmers (21) were unable to detect any influence of electro polishing on

~~face removal and suggest that electro polishing effects are dominant~~

the subsequent tensile behaviour of gold crystals. They however found the flow stress of deformed aluminium crystals lower after surface removal and suggest that electro polishing effects are associated with ^{the} presence of surface oxide film.

Worzola and Robinson (10, 22) observed in silver mono crystals oriented for single slip that by alternately removing a few microns of material from the crystal and then incrementally stressing they were able to double the extent of stage I. However the slope of the stress-strain curve in stage I (Θ_1) was identical to that of a similar crystal strained without intermittent removal. They further observed that the extent of easy glide could be increased if the surfaces were impacted lightly with silicon carbide particles. In stage II however neither intermittent removal nor particle impingement affected the flow curve, Fig. 3

Worzola and Robinson explain these results in terms of the operation of surface sources. If a debris layer is at all formed then sprinkling the surface with silicon-carbide particles would be expected to create additional debris and additional locking of dislocation and this would lead to further hardening rather than increase the extent of easy glide. If surface sources are the ones which operate then polishing would introduce new surface sources by the intersection of grown-in dislocation network by freshly produced surface. Sprinkling the surface would also produce half loop sources

such as observed in ionic crystals. If surface sources are involved in yielding then primary and secondary slip systems should be readily activated leading to a surface layer which is preferentially work hardened relative to the interior of the crystal. Kitajima et al. (12, 13) studied the orientation dependence of preferential surface hardening in copper and α -brass. They found that surface removal in stage II leads to a decrease in the rate of preferential surface hardening presumably related to the reactivation of surface sources and/or a decrease in the near surface dislocation density. They report that for copper crystals deformed in stage I the density of dislocations is higher in the region extending 40 - 70 μ from the surface than in the interior. The geometry of the crystals they used were such that the intersection of primary edge dislocations with $(1\bar{1}1)$ surface would be revealed as etch pits along primary slip directions while slip steps will be observed on alternate pair of faces, Fig. 4. By studying the variation of etch pits distribution as a function of distance from $(1\bar{1}1)$ surface Kitajima et al. consider that the variation in dislocation density is quite opposite to what one would expect if dislocations produced in the interior were to pile up at $(1\bar{1}1)$ surface. So they conclude that instead dislocations are produced at the surface of $(1\bar{1}1)$ by surface sources as shown in Fig. 5. They also consider that the advancing primary glide dislocations interact with the near surface primary and secondary dislocations thereby leading to a dislocation rich layer extending 40 - 70 μ from the surface. This agrees with their estimate of the mean free path of screw dislocations near the surface.

But the results of identical experiments by Block and Johnson (23) show ~~no~~ no dislocation rich layer to a depth of 500 μ . In fact their experiments suggest a decrease in density near the surface followed by an increase towards the interior. The factors contributing to this discrepancy in the results of Block & Johnson and Kitajima et al are not clear.

But it seems that their interpretation of etch pitting results is not unambiguous. Both sets of these data can also be explained by the operation of near surface sources (24). Evidence suggesting this sort of behaviour has been provided by Mitchell et al (25,26).

Despite all these evidences a strong disclaimer for the existence and operation of surface sources is presented by Friedel (27). Specifically if the slip plane on which a potential surface source lies cuts the surface at any angle other than 90° then there is a strong image force inducing the dislocation to cross slip into a position normal to the surface. This then will act as an effective pinning point inhibiting any tendency for preferential operation of the source unless all other sources are heavily locked. Calculations of Lothe show (28) that there exist specific angles of incidence of dislocations for which dislocations can be truly normal approaching the surface. In practical terms metal surfaces are rarely clean and under such conditions the surface sources will normally be pinned. It therefore seems that surface sources are likely to be involved in the yielding process under very special circumstances only.

The results of continuous dissolution of surface of nickel mono crystals are explained by Latansion and Stachle (15, 16) in terms of preferential operation of near surface sources.

Crystals containing 0.1 wt% carbon were deformed in uniaxial tension in 1 N H_2SO_4 under potentiostatic control. Under these conditions the air formed film is quickly removed and surface kept clean. It was found that removal of approximately 3μ of material from the surface immediately before or during elastic deformation resulted in substantial decrease in the value of τ_0 from that of identical crystals deformed in laboratory atmosphere. In addition continuous slow removal of surface increased the extent of stage I while θ_1 was slightly decreased. Likewise stage II was extended and θ_2 decreased, Fig. 6. The slip lines formed while the surface was being actively dissolved, were stronger and more widely spaced than those produced on crystals deformed in air.

Since τ_0 is reduced by surface removal prior to deformation this parameter does not depend on internal sources. This is also not explicable in terms of debris layer hypothesis since this is supposed to form only after yielding although Kramer has recently suggested that micro plastic yielding stress below CRSS may create debris layer which may subsequently be removed by dissolution (30). One factor not in favour of the surface source operation is the observation that the slip lines formed during active dissolution were stronger and more widely spaced than those crystals deformed in air.

By continuous dissolution since the oxide layer and other pinning factors are removed continuously the surface sources must operate more freely and extensively and on many slip lines, in which case both the spacing and intensity of slip steps should decrease.

Latanision and Stachle thus consider that the preferred operation of near surface sources may be the most appropriate mechanism. They site as a supporting pointer the serrated yielding in nickel mono crystals when they are cathodically polarised during deformation. Serrated yielding implies a strong interaction between moving dislocations and solute atoms of hydrogen the point of interest being that at the potential used, 600 mV (SCE), the depth of penetration of hydrogen into the crystal could not be more than 15μ . Sumino's calculations (17) indicate that near surface sources producing loops under their strong image forces can produce large number of dislocation loops than the internal sources. Fabiniak & Kuhlmann-Wilsdorf (31) also point out that the surface acts as a good vacancy sink and hence the near surface region will be depleted of vacancies thus allowing dislocations to be free of jogs and kinks. On the other hand the same region may have an above average concentration of solute atoms. If this layer contributes to a hardening then clearly removal of such a layer prior to deformation will lead to a drop in CRSS (τ_0) as observed by Latanision and Stachle. Obviously solute segregation and vacancy depletion are two opposing factors and near surface sources will act under conditions and at depths where these effects are

optimum. Recently Lohre and Rustad (36) have suggested a dislocation multiplication mechanism operating close to a surface. Zone refined aluminium single crystals were mounted on a lang X-ray camera stressed in a controlled way and photographed in stressed state. From the results they propose that primary dislocation close to the surface cross slip to reduce their line length making anchoring points at which single edged Frank-Read sources may start operating and produce rows or bands of new dislocations.

Another possibility of reduction in τ_0 suggested by Latanision and Stachle envisaged the elimination of back stress on near surface sources arising from a dislocation rich sub surface layer which may exist in undeformed crystals (32). This is distinct from Kramers debris layer and is supposed to arise because image forces can pull grown-in dislocations into ~~distorted~~ regions of the lattice below the surface where they become trapped. The degree to which such a dislocation rich layer might develop would depend on a number of factors including crystal structure, specimen orientation, original dislocation distribution and severity of surface lattice distortion (16)

Fleisher's (35) calculations show that dislocations crossing into a region of different space lattice along the slip direction must create sessile dislocations. Hence the surface itself with its different near surface space lattice will act as a source of surface hardening.

THE CONTROVERSY OVER THE HARD AND SOFT SURFACE LAYERS

In contrast to the above results Fourie (37-41) has reported some very interesting behaviour in mono crystals. In his work slices ranging from 0.03 to 0.6 mm. thick from the surface and interior of mono crystals both as grown and prestrained to various extent, ^{were cut.} The variation of flow stress with the distance of the layer from the surface, Fig. 8, indicates that in crystals prestrained through stage I there is a gradual increase in flow stress for the slices cut from the interior than those from the surface. This is more pronounced at higher prestrains in stage I though at initial stages there is no appreciable difference.

At the onset of stage II the flow stress gradient increases sharply over that which existed in stage I and becomes even more marked in later stages of deformation.

Fourie interprets these results (37-39) in terms of

- (a) operation of dislocations sources throughout the crystal
- (b) A net deficiency in edge dislocations of one sign extending over a distance from the original surface equal to mean free path of edge dislocations. Such a deficiency at the surface would delay onset of stage II hardening in surface regions assuming Hirsch's model for work hardening. Thus a slice taken from the near surface region would exhibit an extensive stage I while one from the interior would exhibit only limited easy glide. One can conclude from this

that the interior of a crystal work hardens more rapidly than the surface regions of a crystal. This is in variance with the conclusion reached by Kramer, Kitajima, Young et al. However Fourié's work was done on specimens thick in comparison to those of other workers so that size contributions may ^{be} relevant (42). Another possible cause for the discrepancy is that dislocation densities and structures of thin foils may not be representative of the bulk material from which the foils were prepared (43) due to dislocation rearrangement and losses during electrolytic thinning. In this point, the rapid relaxation of the surface layer stresses on unloading (6-8) observed by Kramer and accelerated relaxation which occurs with decreasing specimen dimensions (6) imply that foil taken from slices near the surface may well be denuded of dislocations by the time they are examined (44,45). Further the dislocation density measurements by Fourie (38) begin at a minimum distance of 100 μ from the unsliced crystal surface which is just outside the range of thickness of debris layer reported by Kramer. On the other hand all attempts utilizing conventional transmission electron microscopy have also failed to detect any above average density of dislocations in the foils prepared from near surface regions (46-48). Brydes (49) attempted to explain unloading yield point effect on the basis of a soft surface layer formed during first loading portion of the stress cycle. Duquette (50) opines that Fouries and Kramer's works are dissimilar and hence are not contradictory. He proposes a mechanism where surface sources are first involved in plastic deformation and at later stages when these are inactivated by

tangling process the internal sources take over. These then pile up at the surface forming a debris layer. As deformation proceeds the surface dislocations can finally move out while the bulk dislocations cannot. This results in a soft surface and hard interior. Recently Fourie (40,41) from his experiments on copper single crystals seems to have resolved the soft-hard surface layer controversy in favour of the former. In this experiments sub surface regions of copper single crystals which had been strain hardened in tension to the end of stage II was studied by transmission electron microscopy. Sections bounded by original surfaces as well as sections further below surface were investigated. It was found that sub surface dislocation structure was in agreement with flow stress distribution curves which indicate the existence of a soft surface hard core arrangement in strain hardened crystals. Fourie (41) also observed that removal of a surface envelop by chemical polishing a prestrained crystal in the uncondition, reduced the unloading load yield point. He concludes that additional hardening which is introduced during unloading is localised to the surface region. The surface in the stress released state is envisaged to harden with respect to its dynamical state but nevertheless to remain softer than the core.

The formation of a work hardened surface layer is also sensitive to the character of the dislocations passing through the surface. Nakada and Chalmers (33) have worked on aluminium mono crystals which were so oriented, (Fig. 7) that through one pair of sides (S sides)

screw dislocations emerged and from the other pair of faces (E sides) edge dislocations emerged. They found that alternate polishing of S sides and incremental stressing led the crystals to behave in a manner identical to a crystal deformed without polishing. On the other hand crystals whose E-sides were polished behaved in a similar fashion as crystals polished on all sides. They observed these effects only at low strain levels below 2.5% whereas at higher strains this effect is not observed. They conclude that different mechanisms work at lower and higher strain levels. This and other works (18,34) where it was shown that flow curves of F.C.C. mono crystals is especially sensitive to the length of glide path of edge dislocations confirm that surface hardening is associated with emergence of edge dislocations. Thus work hardening behaviour of single crystals is a function of orientation as well as crystallographic relationship between its principal slip direction and surfaces. (FIG. 12)

Effect of Solid Surface Films:

The number of possible ways dislocations react with surfaces will be modified in the presence of solid surface films. As explained in the earlier section three models for the possible modes of dislocation generation were suggested. While in clean crystals it is doubtful whether a surface can block dislocations, the presence of oxide films on the surfaces will block the movement of dislocations. Similarly the surface Frank-Read sources can also be pinned by the

oxide layer on surfaces. Consequently dislocations generated either internally or near the surface will be blocked at the metal-film interface. One possible reason why a solid surface film can block a dislocation is that the substrate and the film have different elastic moduli. Usually a film having higher elastic moduli will repel the dislocations raising its strain energy as they approach the surface. In addition to shear modulus interaction the dislocations may also encounter internal stresses near the region of interface. Some of these effects can be examined in the light of experiments on film effects.

Roscoe's discovery in 1934 that thin oxide films on cadmium mono crystals lead to a significant increase in their yield stress has been corroborated many times by experiments involving electro-polishing to remove oxide layers, electro deposited or evaporated film or alloyed layers. Reviews by Kramer-Demer (51) for metals, Westwood for non metals (52) and others by Machlin (53) Westwood (54) (55), Grosskreutz (56) Benson (57) and Westbrook (56) have clearly documented the influence of films on crystal plasticity. Barretts (58, 59) abnormal effect illustrates dramatically the role of surface films. He found that ^{wires of single} twisted or polycrystals of zinc or iron on immersion in acid lead to a further twist in the original twisting direction. This was attributed by Barrett to the escape of dislocations piled up at the metal oxide interface as the oxide gets dissolved in acid. A more detailed study by Holt (60) indicates that

such an effect is observed only in samples twisted in excess of 10° and is associated with non crystallographic cracks in the oxide film at 45° to the wire axis . Other workers have interpreted fatigue (61,62) creep (63) and tensile behaviour (64-68) of film covered metals in terms of dislocation blocking models. Strengthening effects were observed in metal mono crystals with electrodeposited or evaporated metals (63-65), thin oxide or hydroxide films (61, 62, 66-68). In general such films increase both the yield stress and rate of work hardening and in some cases the three stage work hardening of FCC mono crystals is suppressed (69). Fig. 10 shows the effect of 2 nickel chromium films on the stress strain curve of copper crystals similarly oriented for easy glide. Much of the earlier work of similar nature has been discussed by Andrade (70).

Latanision and Stachle observe (16) that the presence of a $150 - 200 \text{ \AA}$ NiO/NiO_4 passive film on 99.8% pure nickel mono crystals increases the yield stress and produces a generally parabolic stress-strain curve, Fig. 10.

Adams (73) noted that presence of small amounts (1%) of zinc in the surface of pure copper mono crystals produced yield drops. When the zincified surface is polished off the specimens became softer and yield point disappears. .8% zinc at the surface, however increases the yield stress significantly. These results were explained by him in terms of preferential operation of surface sources. Assuming

that slip begins via the operation of surface sources zinc in the surface would raise the CRSS by alloy hardening. However provided the strength of surface layers is not doubled surface sources would still operate. In heavily doped crystals the surface sources would all be locked and other sources would have to be operated raising the yield stress to twice the value of pure copper. Ross (74) observed that CRSS of copper crystals coated with silver was hardly effected but when the crystal was heated to form alloyed layer CRSS increased and work hardening behaviour was same as that for copper alloy crystals containing a low but homogeneously distributed concentration of silver while these are interpreted in terms of the operation of surface sources others feel that (75) they are better explained in terms of preferential operation of near surface sources the point being that surface sources will be locked by surface film or alloyed layer. It was recently reported by Patterson and Greenfield (77) that an alloyed region of 0.4 to 2 μ from the surface created by limited diffusion of Ni and Au to form various surface concentration gradients affected corresponding gradients in elastic constants and lattice parameters. The maximum strength increase was about three times that for pure copper. Rate of work hardening and extent of stage I deformation altered correspondingly.

Grosskreutz has observed (82) that in metals deformed in vacuum (10^{-9} torr) there was a tendency for entrapment of dislocation dipoles just beneath the surface and this tended to reduce

the extent of surface slip band formation. Grosskreutz suggests that modulus of thin film (3000 Å) of Al_2O_3 is increased fourfold under vacuum possibly due to water vapour removal. He observed increase in fracture strength by 50% in vacuum so that reduced tendency for slip band formation at the surface may be both due to elastic repulsion effects and to increased strength of the film. Jemian and Law (83) studied abnormal after effect on polycrystalline wires coated with different metals having elastic moduli lesser than and greater than that of the substrate metals and conclude that the elastic modulus effect is negligible.

Fleischer (29) from his studies, tries to explain the strengthening by oxide films entirely on the basis of the load carrying capacity of the film. The behaviour of the crystal may be described by assuming that the bulk material has a yield stress σ_y and the surface oxide layer an effective strength at yielding of σ_s . For small values of δ one then finds (δ being the effective thickness)

$$\sigma_y = \sigma_y + \delta(\sigma_s - \sigma_y) (A/V)$$

where $A/V = 2(h+w)/h w$, w being width and h the height of the cross-section. He finds that $\sigma_y = 46 \text{ gms/mm}^2$ as the volume yield stress and $\sigma_s = 3 \times 10^6 \text{ gms/mm}^2$ for the effective oxide strength if an oxide thickness of 130 Å is assumed as a reasonable thickness. This result compares well with an effective strength of $2.5 - 3.0 \times 10^6 \text{ gm/mm}^2$ measured by Takamura (95) using samples of varying oxide thickness.

the mismatch

It has been proposed that in the lattices of the substrate and the film also traps the dislocations at the interface. Evans and

Schwarzenburger (84) have pointed that transfer of a dislocation from a substrate of different orientation but the same crystal structure, because of mismatch will produce pile ups at the interface. A film may detach to accommodate slip steps and a polycrystalline film may crack.

Brame and Evans (85) Johnson & Block (76), Ruddle and Wilsdorf (86) have studied effect of thin mono crystal films of various metals evaporated on to a monocrystal substrate of palladium and silver such that film and substrate lattices are in parallel orientation. Any mismatch between lattice should be accommodated by a two dimensional network of dislocations Fig. 14 . Effectiveness of this network as a barrier would diminish for smaller lattice mismatch. By transmission electron microscope study it was seen that dislocations transfer indeed occur for misfits around 3% but blocked at larger misfits. Ruddle and Wilsdorf (86, 89, 90) have reported that oriented copper mono crystals coated with 600 Å epitaxial electrodeposit of nickel yield in tension at approximately half the stress required for unplated crystals Fig. 15 . They interpret their results in terms of theoretical predictions of van der Merwe (87) and Jesser and Kuhlmann-Wilsdorf (88) that for lattice misfits of 4%, an electro deposited film will be elastically strained to match the substrate lattice at least in the early stages. Johnson and Block (76) demonstrate that the magnitude of strengthening effect cannot be explained in terms of lattice mismatch alone. For example an electro deposited gold coating strengthens copper and though the mismatch is 11.3% the gold film behaved in a ductile manner. Epitaxial rhodium also produced strengthening and though the mismatch was only 4.5% exhibited

brittle cracking. Epitaxial copper with zero mismatch also produced strengthening.

Block and Johnson (76) earlier had reported that there existed a perturbation of flow behaviour of a crystal which had been plated and stripped as compared to an unplated crystal and attribute this to subsurface damage by the plating. They explain this by saying that film cracking either prior to or during deformation may create stress pulses and these inject dislocations into the substrate. But Fridman et al (78) attribute this to the fact that these two experiments were done on two different crystals, the difference in flow stresses being well within experimental errors for two samples. Instead Fridman et al have done experiments on the same sample to eliminate the ambiguity. Copper single crystals were prestrained with no film unloaded and plated. Even when no cracks were found there was an increase in flow stress. In another case when coating was applied and removed at similar stage the deformation behaviour was unperturbed. They conclude that damage zones have negligible effect the reason being, other zones covering an area 99.5% of bulk volume remain unaffected by damage zones.

Mention must be made regarding the Rebinder effect, electrocapillary effect, liquid metal embrittlement and stress corrosion cracking. Though obviously related to surface effects the details of these are beyond the scope of this investigation.

~~Abstract~~: In the present study, the effects of anodic oxide films on the plastic deformation of aluminum single crystals have been studied. It is clear from the previous studies that the edge and screw dislocations play different roles in the strain hardening of metal crystals with relatively clean surfaces. One of the aims of the present investigation has been to obtain further evidence for the differences in the parts played by edge and screw dislocations in the strain hardening by selective oxidation of certain surfaces of aluminium single crystals. Further it is hoped to understand the mechanism of hardening in crystals when oxide layers are present on the surfaces.

EXPERIMENTAL PROCEDURE

It consists of growing aluminium single crystals, cutting them to suitable sizes, determination of orientation by X-ray method, preparation of tensile specimens by suitable techniques and mechanical testing.

CRYSTAL-GROWTH: Modified Bridgmann technique was used, Fig. 15, to grow the crystals in a graphite mold. The method is a modification of zone refining technique and it essentially involves directional solidification. The metal in solid state is held in a crucible and a molten zone is allowed to traverse through the length of the material starting at the bottom tapered to a sharp - V. At a suitable rate of traverse of the hot zone only one nucleus forms at the bottom of the crucible. Hence as the top is reached generally a single crystal is left behind. An internally wound resistance heated furnace was used for this purpose. A coil of a 16 gage Kanthal wire of grade "A" was wound on a dummy, a cardboard tube of 3" outer diameter. Over this, high alumina cement (Fire Creet) mixed into a thin paste with water was evenly applied such that the coils were $1/8$ " below the cement skin. This was allowed to dry in air for 3 days and then the coils were connected to a regulated power supply. Power was adjusted to get a temperature of 150° and the furnace was allowed to remain in this state for 2 days, The temperature was then raised to 400°C and the furnace allowed to remain at this temperature for another 2 days.

This slow and gradual heating was to ensure a gradual and complete removal of moisture since rapid heating in the beginning causes the cement to crack. The furnace was then allowed to heat to a high temperature and the cardboard dummy burnt off. Then this whole assembly was fixed in an aluminium shell of 6" diameter with the ends closed and fixed by asbestos end plates. The space between the cement and shell was filled with asbestos powder. The furnace power rating was adjusted to a suitable value knowing the resistance and other necessary parameters from the Kanthal-handbook.

In the early stages of crystal growth, troubles were encountered due to faulty mold design as well as inadequate atmosphere control. In the first place high purity aluminium pellets were used. It was found that due to inadequate atmosphere control the surface of the pellets rapidly oxidized. This prevented the molten metal inside the pellets to coalesce. After cooling the pellets remained as pellets with a thick oxide coating around. This behaviour persisted even at a temperature as high as 900°C in the crucible. As a remedy a gas purification train was used to purify argon gas. But effective plugging of leaks was found to be extremely difficult.

It was then decided to evacuate the tube using mechanical pump throughout the period of crystal growth. But time and material availability were factors which prevented the construction of such a unit. Prior to letting in argon and heating, the tube was evacuated for 1 hour using rotary pump. But even by this procedure the pellets did not melt and flow down. Purified argon was used at this stage and a two stage melting was attempted. Argon was sent through a gas train consisting respecti-

vely of Keklers reagent to remove oxygen, NaOH, CaCl_2 to remove the moisture, copper turnings heated to 400°C to remove CO_2 and titanium pellets heated to 900°C to absorb oxygen. It then passed through diffusion pump oil into the system. Pellets were ~~kept~~ in the spherical seed portion and the top portion and were seperately heated to cause melting. A partial melting could be achieved but the metal again did not flow down the stem. It was decided to cast the seed and the stem seperately. Air melting was tried successfully but this required a preheating of the mold which severely eroded the mold walls. Finally the seed was successfully cast in a vacuum induction melting unit. The stem portion was cut from a sheet rolled from a piece of aluminium cut from an ingot of high purity metal. They were cut into strips of suitable dimensions and filed such that they could be placed easily in the mold slot. After effecting some modifications a 3 channel mold, Fig. 20, was finally used and 3 crystals, each of $1/8''$ x $1/4''$ cross section and 6" length were grown from a single seed at a time. First a single crystal seed of spherical shape was grown and the stem portion cut off. This was then oriented in the desired direction using Back Reflection Laue method of X-ray analysis. The desired direction was obtained and the spot marked. This was then placed in the mold cavity with the marked spot at the stem slot. By trial and error the temperature of the furnace, the position of the hot zone and time were adjusted for a partial melting of the spherical single crystal seed. The furnace was then raised slowly and hot zone allowed to reach the top. The rate of the zone travel was adjusted to be 1" per hour and the maximum temperature of the furnace was 900°C . The furnace was then cooled down by reducing the voltage and when the furnace cooled down to 400°C an annealing run was made,

Even at this stage the oxidation problem could not be completely

eliminated in the absence of a perfect leak proof gas purification train. The result was an improper joining of the seed and the stem. This was due to two reasons. The oxide layer formed on the seed as well as the stem bottom prevented joining. The stem itself having an oxide layer around gets stuck to the mold walls rendering the non-wettable nature of graphite useless. This led to pronounced surface irregularities as well. This problem could be solved partially by placing a weight over strips placed in the slot which protrude over the top. Thus as soon as there is a partial melting in the seed the stem is pushed down into the melt, breaking the film. But this difficulty can be overcome only in a high vacuum or a leak proof system with highly pure inert gas.

Crystal Cutting: and Orientation determination in Back Reflection Lane

Method: Crystals were cut free of strain using an acid crystal cutter. The crystals attached to the seed was gently transferred to the crystal holder and mounted firmly with molten paraffin wax. The stem was then cut off from the seed and the crystal cut into pieces 4 cms. in length. The acid needed was a mixture of 30% HF and dilute HCl.

The crystals were then mounted on a goniometer and using back reflection Lane method the orientation was determined. Tungsten radiation was used and an exposure of 1 hour was enough to get sufficient number of spots.

Preparation Of Tensile Specimens:

Polishing: Electro polishing was used with advantage to get the required specimen shape, a sort of electro machining, and to get a mirror bright polish.

Crystals were etched in dilute HCl or dilute HF and washed with

distilled water. They were then dipped in dilute NaOH solution to remove any grease. An initial chemical polishing was tried in various acid mixtures consisting of nitric, hydrochloric, orthophosphoric and acetic acids. Though they gave a bright surface, polishing conditions were not properly controllable. Moreover the surface irregularities with quite pronounced and deep depressions or ridges had a tendency of getting attacked vigorously and to become deeper and wider. So it was decided to go for electropolishing straightaway. The best electrolyte was found to be a mixture of perchloric acid and acetic anhydride mixed in the proportion:: 345 ml. : 655 ml. (94). This mixture has a danger of being explosive above 30°C . So adequate precautions have to be taken to cool it. The cathode, a small aluminium or stainless steel vessel containing the solution was cooled externally in ice. To get a fine polish the specimen was rotated in the electrolyte at around 100 rpm. This was connected to the anode of the power supply through graphite bushes.

It was seen that under normal polishing conditions the polishing was not at all satisfactory. To polish a specimen in the plate ^{area} region an initial degree of surface smoothness is required. A fairly good mechanical polishing would be necessary initially to get a good electro polish under normal polishing conditions.

But the nature of the specimen and its shape and size forbade any mechanical polishing. Under normal electro polishing conditions the polishing being extremely slow, even after hours of polishing the surface ridges could not be removed. They tend to get deeper due to preferential attack, and widen out. They persisted even after removal of nearly $\frac{1}{4}$ " thick material, though a bright surface was obtained.

It was observed that above the plato region under high current conditions the ridges were not attacked preferentially, the surface was dissolved rapidly and the ridges started disappearing. But the specimen was rapidly thinned down and the solution gets heated up in a matter of a fraction of a second. This is dangerous since the electrolytic mixture above 30°C has a reputation of being as explosive as TNT. Moreover the specimen cannot be allowed to get thinned down too much. Also the chucks were designed to grip a specimen 1/8" thick. So 1 cm. on either side was masked off by a suitable insulating material leaving a small portion at one end for the gripping in a clip. Shellac dissolved in methanol was tried. But this peeled off fast. It was found that a thick coat of epizone wax dissolved in tri chloro ethylene, after ~~dry~~ing, was very effective. Further trouble was encountered due to rapid heating of solution at the edges of wax coating. The specimen was etched deep at this junction and the portion on either side formed necks so that the cross section at these places was well below that in the gage portion, thus rendering the specimen useless for testing.

At this stage aluminium cathode was used and under low voltage (below the plato region) conditions as well as high current conditions aluminium from the cathode as well as anode dissolved in large quantities and the resistivity of the solution dropped rapidly. At a certain stage it was as low as 10 - 12 Ω (ohms). When polishing in this solution was tried under high current conditions at 8 - 10 amperes, some of the earlier difficulties were overcome entirely.

(a) The ridges were not etched deep. On the contrary they were not attacked due to an overall rapid attack and removal of surface layers. This led to the obliteration of the ridges rapidly. This solution did

not get heated fast and allowed for a polish of 3 - 4 minutes at a stretch.

(b) At the edge of the masked regions no preferential attack took place. In fact there was a taper as is obtained on standard machined specimens.

(c) Throughout the length of the gage there was uniform attack.

The resulting surface was fairly smooth but not bright. After polishing this in a fresh solution for just over 10 minutes a mirror bright finish was obtained. This specimen has the advantage of having a thinner section at the gage portion as compared to the grip portion thus eliminating the grip problems.

Anodising: Wherever necessary anodizing was done in a solution of tartaric acid and ammonia adjusted to 5.5 pH. The thickness of the oxide layer is given by the standard voltage-thickness relationship for this limiting or barrier film technique. For this solution 1 volt gives an oxide layer 13.5 \AA° thick. This is one of the solutions that gives a very compact non porous oxide film and it is for this reason the solution was preferred. A milli ammeter was connected in series and power supply knob turned slowly such that the current does not shoot up above 25 m.A. It takes 2 - 3 minutes to reach 80 V under such conditions and the current gradually falls back to zero. Strictly speaking the current does not fall to zero but reaches a very low and stable value which is called the leakage current. At this stage power supply was cut off and the specimen taken out and thoroughly washed in distilled water. The cathode used was an aluminium vessel which holds the solution.

Mechanical Testing: Experiments carried out mainly consisted of testing specimens at room temperature and at 0°C in tension on INSTRON machine.

The gage length, width and thickness of the specimens were measured accurately prior to testing. The grips used for tensile testing were made of brass and is shown in Fig. 17a. Fig. 17 b shows the outer jig assembly. The specimens were mounted, keeping the grips in the casing. The grips were held in position with a top cover fixed on to the casing with screws. The whole assembly was then mounted on to the machine and the outer covering removed. This procedure was followed carefully throughout to ensure that no undue pressure or damage is inflicted on the single crystals while handling. The scheme of experiments is given in table number: 1.

SCHEME OF EXPERIMENTS:

The table given below shows the scheme of experiments, the first six with specimens of preferred orientation (Fig. 20), the last ten randomly oriented specimens. Fig. 19 shows the relative orientation of all the crystals.

Unless otherwise mentioned all surface and oxide dissolutions ~~was~~ ^{were} done by electro polishing the specimen. In cases where the amount of material removed by electropolishing is required it is indicated. Here dissolving $2w = x$ microns means total material removed from the width section (on either side) is x microns. Likewise $2t = y$ microns denotes a total removal of y microns from the thickness of the specimen. This difference in the rate of metal removal from width and thickness is due to the rectangular cross sectional shape of the specimen as well as due to small eccentricities in the rotation of the specimen.

Anodizing was always done at 80V amounting to an oxide thickness of 1080 \AA° from the standard $1 \text{ volt} = 13.5 \text{ \AA}^{\circ}$ relationship for the tartaric acid ammonia solution at 5.5 pH.

In some of the oriented samples during surface dissolution either the opposite broad faces or thin faces were masked off. In such cases the amount of material removed respectively from thin faces or broad faces is given in appropriate notation. Throughout a strain rate of 0.01/minute was used. The gage length of the specimens tested was 2 cms.

TABLE I

Specimen Number	Nature of the Test	Steps involved in the Test	Figure No.
1	To study the effect of oxide layer on S-sides and the intermittent dissolution of S-sides as well	a. S-sides anodized at 80V b. Dissolved $2t=33.5 \mu$ c. Dissolved $2t=22.5 \mu$ d. At room temperature for 10 hours e. Dissolved $2t=187.5 \mu$ f. Dissolved $2t=320 \mu$	No. 21
2	Effect of oxide layer on the E-sides and the effect of intermittent dissolution of E-sides	a. E-sides anodized at 80V b. Dissolved $2t=27.5 \mu$ c. Dissolved $2t=15 \mu$ d. At room temperature for 10 hours e. Dissolved $2t=292.5 \mu$ f. Dissolved $2t=57.5 \mu$	No. 22
3A	Surface dissolution tests on E-sides leaving S-sides masked to avoid dissolution	a. Clean crystal b. Dissolved $2t=57.5 \mu$ c. Dissolved $2t=110.0 \mu$ d. Dissolved $2t=217.5 \mu$ e. Dissolved $2t=62.5 \mu$ f. Dissolved $2t=212.5 \mu$ g. Dissolved $2t=77.5 \mu$ h. Dissolved $2w=47.5 \mu$	No. 23
3B	Surface dissolution test on S-sides leaving E-sides masked	a. Clean crystal b. Dissolved $2w=47.5 \mu$ c. Dissolved $2w=100 \mu$ d. Dissolved $2w=165 \mu$ e. Dissolved $2w=47.5 \mu$ f. Dissolved $2w=107.5 \mu$ g. Dissolved $2w=72.5 \mu$ h. Dissolved $2t=42.5 \mu$	No. 24

Specimen Number	Nature of the Test	Steps involved in the Test	Figure No.
4A	Surface dissolution test starting with a crystal the E-sides of which are anodized at 80V. Anodizing is done at other stages as well followed by dissolution	a. E-sides anodized at 80V b. Dissolved 2t=70 μ c. Dissolved 2t=285 μ d. E-sides anodized at 80V e. Dissolved 2t=524 μ	No. 25
4B	Surface dissolution test starting from a crystal the E-sides of which are anodized at 80V. Anodizing is done at other stages as well followed by dissolution	a. S-sides anodized at 80V b. Dissolved 2w=26 μ c. Dissolved 2w=107 μ d. S-sides anodized at 80V e. Dissolved 2w=43 μ f. Dissolved 2w=64 μ	No. 26
5-A	Tensile Test at 0°C.	crystal tested clean	27
5-B	Tensile Test at 0°C	crystal tested after anodizing at 80V	
6-A	Tensile Test at 0°C	Same as 5-A	28
6-B	Tensile Test at 0°C	Same as 5-B	
7	Tensile Test at room temperature with intermittent surface dissolution	a. Clean crystal pulled in tension b. Dissolved 2w=40 μ 2t=55 μ c. Dissolved 2w=62 μ 2t=55 μ d. Dissolved 2w=117 μ 2t=74 μ e. Dissolved 2w=320 μ 2t=190 μ f. Dissolved 2w=107 μ 2t=45 μ g. Dissolved 2w=151 μ 2t=78 μ	29

Specimen Number	Nature of the Test	Steps involved in the Test	Figure No.
		h. Dissolved $2w=457 \mu$ $2t=198 \mu$	
8	Tensile Test starting from an anodized crystal followed by intermittent dissolution of surface	a. Crystal anodized at 80V b. Dissolved $2w=54.5 \mu$ $2t=24 \mu$ c. Dissolved $2w=128 \mu$ $2t=44 \mu$ d. Dissolved $2w=432 \mu$ $2t=114 \mu$ e. Reanodized at 80V f. Dissolved $2w=83 \mu$ $2t=58 \mu$	30
9	Intermittent surface dissolution in 3% NaOH	a. Clean crystal b. Kept 2 minutes in 3% NaOH c. Kept. " d. Kept 5 minutes in 3% NaOH e. Kept 10 minutes in 3% NaOH	31
10	Intermittent surface dissolution and anodizing done alternately	a. Clean crystal b. Anodized at 80V c. Oxide layer removed d. Anodized at 80V e. Oxide layer removed f. Reanodized at 80V g. Oxide layer removed h. Reanodized at 80V i. Oxide layer removed j. Reanodized at 80V k. Oxide layer removed	32
11	Intermittent surface removal starting from clean crystal	a. Clean crystal b. Dissolved $2w=8 \mu$ c. Dissolved $2w=12.5 \mu$ $2t=12.5 \mu$	

Specimen Number	Nature of the Test	Steps involved in the Test	Figure No.
		e. Dissolved 2w=90 μ 2t=53.7 μ	
		f. Dissolved 2w=20 μ 2t=17 μ	
		g. Dissolved 2w=20 μ 2t=25 μ	
		h. Dissolved 2w=12.5 μ 2t=12.5 μ	
		i. Dissolved 2w=67 μ 2t=40 μ	
		j. Dissolved 2w=85 μ 2t=50 μ	
		k. Dissolved 2w=13 μ 2t=7 μ	
		l. Anodized at 80V	
12-A	Crystal anodized after stressing it to a predetermined level	a. Clean crystal b. Anodized at 80V	34
12-B	Crystal anodized and oxide layer stripped off at the stress level at which above crystal was anodized	a. Clean crystal b. Anodized and oxide layer stripped off	
13	Tensile test at room temperature starting from an anodized crystal and then reanodizing it at regular intervals	All steps from a. to g. involves anodizing specimen at 80 V	35
14	Stress relaxation test in air as well as in 3% NaOH	a. Stressed to a level of $\sigma = 0.8145 \text{ Kg./mm}^2$, $\epsilon = 0.177 \text{ Kg./mm}^2$ b. Stressed in air b. Stress Relaxation in air c. 3% NaOH applied d. Washed in distilled water e. Dried in jet of acetone f. Reloading in air	36

Specimen Number	Nature of the Test	Steps involved in the Test	Figure No.
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g. Stress relaxation in air

h. 3% NaOH applied

RESULTS AND DISCUSSIONS

CRYSTALS WITH PREFERRED ORIENTATION (CRYSTAL NUMBERS: 1, 2, 3A, 3B, 4A, 4B)
(FIGURES: 1 - 6)

These crystals were oriented as shown in Fig. 20. No.1 was oriented as in 20-b and the rest as shown in 20-a. The sides through which edge dislocations emerge out predominantly are termed as E-sides and the sides through which screw dislocations emerge out predominantly are termed as S-sides.

Figures 21 and 22 show the effect of oxide layer on the s-sides and E-sides respectively. The stress at which the specimen no. 2 yields 0.6 Kg./mm^2 is greater than that for no.1 (0.48 Kg./mm^2). But the slope of the ~~stress~~_{strain} curve for specimen (1) is greater than that for specimen no. 2.

Due to non availability of more than one specimen of the type no. 1 all the following tests were carried out on specimens of the type no.2. So for ~~these~~ crystals E and S sides had different areas, E sides having approximately double the area of S-sides. For these crystals surface ~~dissolution~~ tests combined with anodizing were done intermittently

Figs. 23 and 24 show the effect of dissolution of E sides and S sides respectively on specimens: 3A and 3B. Dissolving surface layers from these crystals below a critical strain raised the stress level considerably. This effect was observed in all other specimens both randomly oriented and those with preferred orientation. Dissolving approximately 25 microns from the E faces as well as S faces raised the stress levels approximately to the same extent.

were dissolved beyond a strain of 0.05, very little drops in stress levels were observed, in 3A when E sides were dissolved the drop was marked. There was a drop at a strain of 0.025 itself as shown in step-d, but a further deformation and subsequent dissolution once again produced a rise in stress level at 0.05 strain and thereafter dissolution resulted in drops.

When S-sides were dissolved in 3-A there was no effect as seen from step-h. But a dissolution of E-sides in 3-B produced a marked drop in step-h.

There is an overall softening effect for the specimen 3-A whose E-sides were dissolved as compared to 3 B, the S-sides of which were dissolved except in step h. This is clear from the stress/strain curves. For specimen 3A for a strain level of 0.125 the stress level is 1.12 Kg./mm.² in fig. 23 and for specimen 3B at the same strain the ^{stress} level is 1.5 Kg./mm.² (Fig. 24). ^

Figures 25 and 26 show the effect of anodizing and dissolution on the E sides and S-sides for specimens 4A and 4B respectively. Anodizing E-sides in 4A shows a higher yield stress initially (0.64 Kg./mm.²) as compared to 4B the S-sides of which were anodized (0.55 Kg./mm.²). This is in accord with the observation for specimens 1 and 2. Further dissolution of S-sides in 4B continued to show small rises in stress level upto a strain of 0.10 and thereafter small drops whereas anodizing had no effect whatsoever as seen in step-d, Fig. 26.

But dissolution of E sides after a strain of 0.01 caused the stress level to drop appreciably as seen in steps c and e of 4A. Ano-

step d, fig. 25. As in 3A and 3B there was an overall softening effect for 4A the E-sides of which were involved in anodizing and dissolution treatment as compared to specimen 4B the S-sides of which were involved in dissolution treatment. This is apparent when the stress levels of 4A and 4B are compared at a strain of 0.04.

In the case of aluminium, the surface is always covered with a thin natural oxide layer, the properties of which are not clearly known. An anodized crystal has a surface oxide layer thicker than the above mentioned natural oxide layer. Generally an oxide layer on the surface would block the dislocation egress whatever be the mechanism by which they are generated. Thus they would pile up at the oxide-metal interface and the crystal may be strengthened at a rapid rate. From Figs. 21 and 22, the yield strength of the crystal whose E sides were anodized (0.6 Kg./mm.^2) is seen to be higher than that of the crystal whose sides S-sides were anodized (0.48 Kg./mm.^2) (4). Though a comparison of stress values in two specimens is not strictly valid, the higher yield stress of specimen no:2 may be due to the reason that the surface oxide layer is expected to be a more effective barrier in the case of edge dislocations than for screw dislocations. The fact that for crystals 4A and 4B both of which were cut from the same parent crystal no.4, the yield stress of specimen no.4A whose E sides were anodized is higher than that for specimen 4B whose S-sides were anodized corroborates the above conclusion.

The work hardening behaviour is also sensitive to the glide path of edge dislocations as seen from Fig. 21 and 22. The crystal no.-1 work hardens at a faster rate as compared to crystal no:2. The glide path of edge dislocation in crystal 2 is approximately half the glide path of edge dislocations in crystal no:1 and this leads to the lower

rate of work hardening in crystal no:2 Figs: 22 and 21 . One would expect higher rate of work hardening in crystal no:2 since the E sides of this crystal are anodized leading to a rapid pile up of ^{edge} dislocations at the metal-oxide film interface whereas for crystal no:1 the screw dislocations would not be much affected by the oxide layer on S-sides: But since this does not seem to be the case one can conclude that work hardening rate is sensitive more to the glide path of edge dislocations than to the film barrier effect.

The results of surface as well as intermittent anodizing also establish the different roles the edge and the screw dislocations play in the hardening process. The initial rise in stress level when surface layers were dissolved intermittently below a critical strain level is one which is observed even in crystals which are randomly oriented. This will be discussed separately in the later sections; but it is to be noted that surface dissolution from both E and S sides lead to this type of behaviour and probably the mechanism of work hardening is different at lower strain levels as compared to strain levels above 0.05. Intermittent surface dissolution of S-sides carried out for specimen 3B Fig. 24 and specimen 4-B Fig. 26 resulted in small drops in stress levels whereas equal amounts of dissolution from E-sides in specimens 3A Fig. 23 and 4A Fig. 25 at identical levels produced greater drops in stress levels. This shows that edge dislocations are effectively blocked at the surface and get piled up thereby leading to a work hardened surface layer where the dislocation density is possibly higher than in the bulk. When such a layer is dissolved the bulk becomes softer and hence a fall in the stress level. Whereas the screw dislocation having a greater amount

of freedom can cross slip and get annihilated when they encounter screws of opposite orientation. So the pile up of screw dislocations at S-faces will not be as significant as that of the edge dislocations and hence lead to smaller drops in stress levels when the S-sides were dissolved as seen from Fig. 24, 26. This is thoroughly confirmed from steps-h of Figs. 23 and 24. Whereas in steps a to g the E-sides of the crystal 3A were dissolved in step h the E-sides were masked off and the S-sides were dissolved. This leads to no drop in stress level whatsoever. Likewise for crystal 3B in Fig. 24 in step-h the E-sides of it were dissolved leading to a significant drop while in all other steps the S-sides were dissolved and show no significant drop. Similarly Figs. 25 and 26 for 4A and 4B show the effect of intermittent anodizing. In Fig. 25, step d when the E-sides were anodized for specimen 4A there is a significant rise in stress level while in Fig. 26 step-d anodizing S-sides for specimen 4B had no effect. In these crystals intermittent dissolution of E-sides lead to a general softening of the specimen as compared to the dissolution of S-sides. These observations are in accord with the results obtained in similar experiments by Nakada and Chalmers.

RANDOMLY ORIENTED SPECIMENS:

The effect of the surface oxide layer is shown in Figs. 27 & 28 for specimen nos. 5 & 6. For each orientation two specimens were tested one with an anodized oxide layer 1080Å thick and another after electro-polishing. In general the yield stress of specimens which were anodized was higher than that for specimens tested without the benefit of an anodizing treatment. While for specimen no. 6 the strengthening effect persists to higher strains, for specimen no. 5 the two curves are almost identical beyond 0.07. This difference is due to the difference in orientation for the two crystals. Specimen no. 6A showed a 3-stage work hardening behaviour, while in 6B when tested after anodizing this was suppressed.

When a tensile test was interrupted and the sample was anodized, on restraining a marked strengthening effect was observed. Such tests were done upto about 0.07 tensile strain only. This is clear from step e of Fig. 30, steps b,d,f,h of Fig. 32, step l of Fig. 33 and step b for crystal no. 12-A of Fig. 34. But when initially anodized samples were interrupted at strains beyond 0.06 and reanodized the flow stress was observed to decrease (Fig. 35 steps a to f). The effect of surface dissolution at low strains (below 0.05 to 0.06) can be seen in step b of Fig. 29, step b of Fig. 30, step b of Fig. 31, steps c,e,g,i,k of Fig. 32 and steps b to i of Fig. 33. This behaviour is observed for different amounts of surface dissolution. In Fig. 33 it is seen from steps g,h,i that the slope of stress/strain curve falls rapidly on stressing after a surface dissolution treatment. Also surface dissolution following anodizing and stressing caused an increase in flow stress.

But dissolving surface layers intermittently above a strain of 0.05 to 0.06 always lead to drops in flow stress. This is clear from steps c to h of Fig. 29, steps c, d, f of Fig. 30, step e of Fig. 31 and steps j and k of Fig. 33. But surface dissolution greater than 200 μ did not effect proportionately greater drops in stress levels. Whenever large amounts of material was dissolved, the stress values were corrected to compensate for the drop due to reduction in cross sectional area.

To study the effect of any sub surface damage during anodizing crystal no., 9A was anodized after interrupting the test at a pre determined stress level and deformed further. Crystal 9B was anodized and oxide layer stripped off after interrupting the tensile test at the same stress level at which anodizing was done for 9A. While for 9A a strengthening was observed, for 9B on further deformation an abnormal increase in flow stress was observed. This is shown in step b for both 9A and 9B in Fig. 34.

Fig. 36 shows the results of stress relaxation test in air as well as in 3% NaOH. Upto step b and from g to h stressing was done in air and from b to c as well as from g to h the specimen was allowed to undergo stress relaxation in air. At c and h, a 3% NaOH bath was applied and seen from the figure there was a sudden drop in stress level.

It should be noted here that Takamura reports yield stresses for 99.99% pure aluminium that ranges for 100 to 300 gm/mm^2 . But in our case generally the yield stresses are higher (around 500-600 gm/mm^2) which may be due to higher amount of impurities. It will also be appropriate to take note of Takamura's work with different electro polishing

baths. The thickness of the oxide layer left over after polishing with Jaquet's bath (perchloric acid + acetic anhydride) was found to be around 100 \AA while that left over after polishing in orthophosphoric acid baths was around 500 \AA . He found that the thicker oxide layer had a greater strengthening effect. But the strength properties did not differ much for specimens electropolished in Jaquet's bath and ^{for} those with a natural oxide layer formed after the surface oxide layer was removed by etching in dilute HCl. The thickness of this naturally formed layer was found to be 50 \AA .

Of the various mechanisms suggested in literature to explain the film strengthening effect viz., Fleischer's model of strengthening, film barrier for dislocation egress, locking of surface sources, sub surface damage and residual stresses, the film blocking of dislocation egress leading to a pile up appears to be the more attractive. Whatever be the mode of generation of dislocation they will be expected to pile up at the metal/oxide interface. The instantaneous drop in stress level when a 3% NaOH bath was applied to a specimen undergoing stress relaxation in air lends credence to the above supposition. This ^{twisted} is similar to Barret's abnormal effect in specimens. The dislocations piled up at the metal /oxide interface move out of the crystal when the oxide layer is dissolved by the NaOH bath.

The results of surface dissolution tests conducted intermittently starting either from a clean crystal or anodized crystal above a strain of 0.05 to 0.06 are also in general agreement with the supposition that dislocations pile up at the surface forming a dislocation rich layer. When this layer is dissolved naturally the flow stress decreases.

A result that cannot be explained in terms of the oxide layer acting as a barrier to the egress of dislocations is the following. When an anodized specimen is further strained and the test interrupted and the specimen reanodized the cracks in the oxide layer will heal up and this should normally strengthen the crystal. But such a treatment actually lead to a drop in flow stress. This may possibly be due to the reason that local heating that may occur during anodizing at the high strain levels involved (above 0.06) may cause some annealing thereby leading to the softening observed.

Experiments were conducted to see whether anodizing causes sub surface damage thereby contributing to the strengthening effect. In crystal 12 A when anodizing was done after interrupting the tensile test a normal strengthening was observed on restraining. But when another crystal of same orientation (12 B) was anodized and oxide layer stripped off immediately thereafter, an abnormal strengthening was observed. If this were to be due to sub surface damage or residual stresses, then understandably the rise in stress level should be less than that for 12A with an oxide layer. This and the treatment being well below the 0.06 strain level points to the phenomenon what may be termed as "surface removal effect at strains below 0.05 to 0.06".

The increase in flow stress following a surface dissolution below a strain of 0.06 may be due to removal of a soft surface layer formed during deformation as observed by Fourie in copper single crystals. But the natural oxide layer on aluminium should normally block the dislocation egress thereby stopping any soft surface layer formation. Putting a thicker oxide layer by anodizing leads to a strengthening

and dislocations are expected to pile up at the interface and dissolving surface layers should lead to a drop in flow stress. But even this lead to an increase in the flow stress. One other possibility of explaining this anomaly is in terms of solute locking of dislocations. At the low strain levels solute atoms of small impurities present migrate to dislocation sites under the unloaded condition. This pins down the dislocations in the dynamic state and leads to a hardening.

CONCLUSIONS

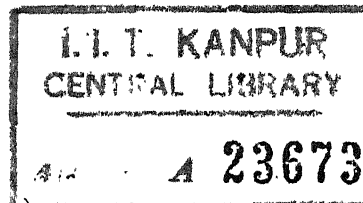
- (i) Anodized oxide layer causes an increase in the strength of aluminium crystals. This is likely to be due to the oxide film acting as a barrier for dislocation egress.
- (ii) Dissolution of surface layers beyond a strain level of 0.05 to 0.06 leads to a drop in flow stress. The depth of dissolution upto which this softening is observed is 200 microns.
- (iii) Below about 0.06 strain surface dissolution leads to an increase in flow stress in contrast to that observed at strains beyond 0.06.
- (iv) Reanodizing a crystal following an initial anodizing and stressing lead to a drop in flow stress on further deformation in tension, beyond a strain level of 0.06.
- (v) The blocking effect of oxide films is more effective with edge dislocations than with the screw dislocations. The yield stress of crystals, the edge faces of which were anodized is higher than that for crystals, the screw faces of which were anodized. Anodizing edge faces after the crystal has been stressed initially raises the flow stress whereas the flow stress of crystals, the screw faces of which were anodized after an initial stressing, remains unchanged. Likewise dissolving the screw faces at higher strains had no effect on flow stress while dissolving the edge faces produced a marked drop in flow stress.
- (vi) The glide distance of edge dislocations is more important in governing the work hardening rate of single crystals.

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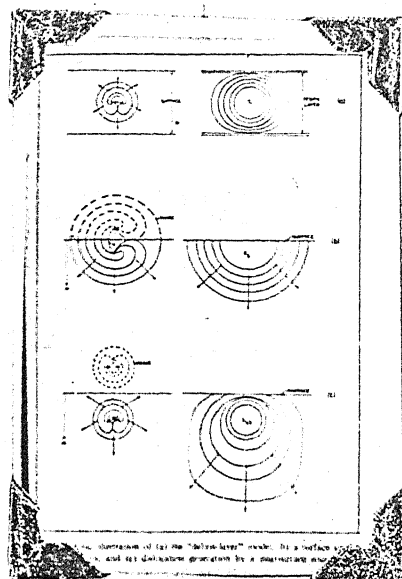


FIG. 1

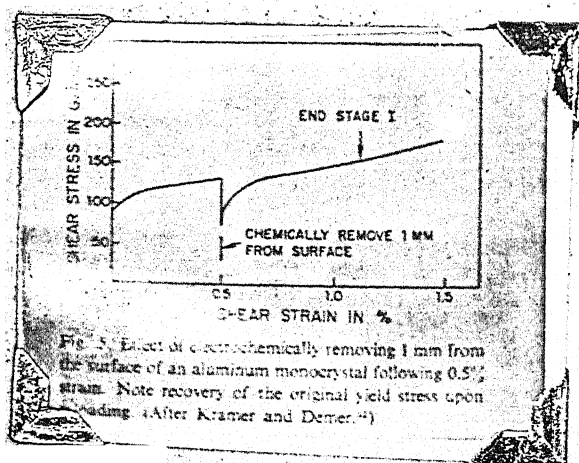


FIG. 2

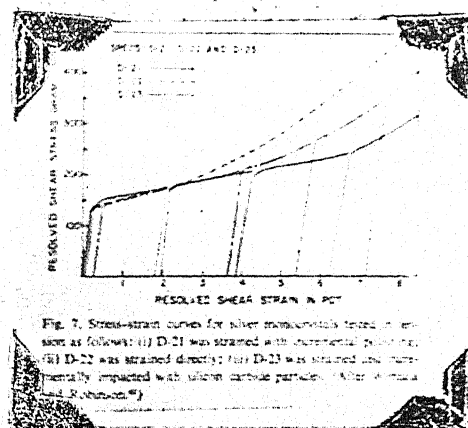


FIG. 3

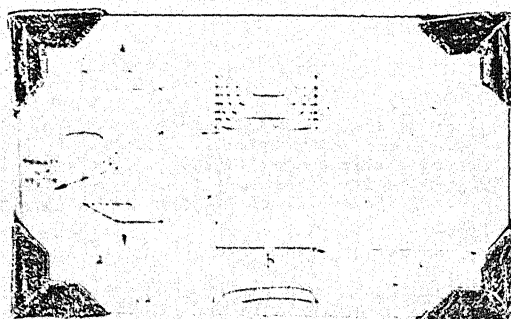


FIG. 4

FIG. 5

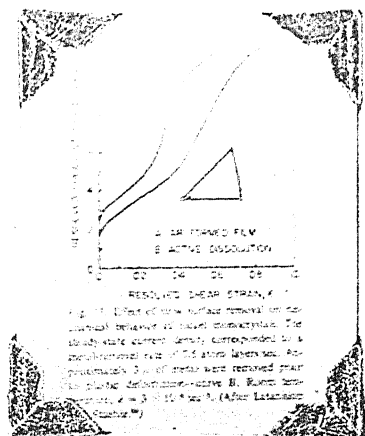


FIG. 6

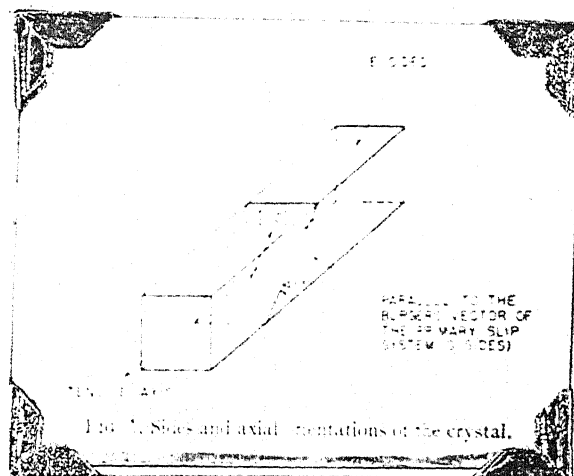


FIG. 7

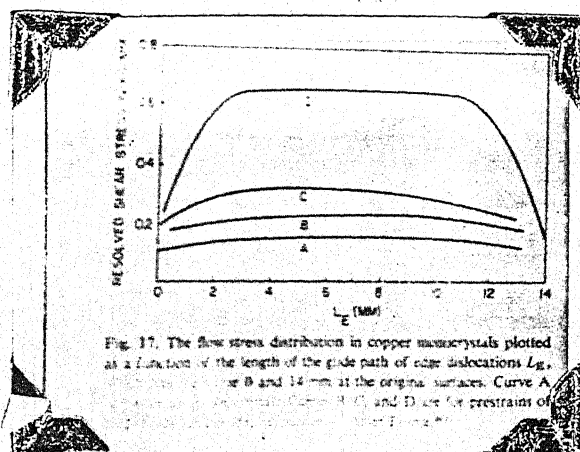


FIG. 8

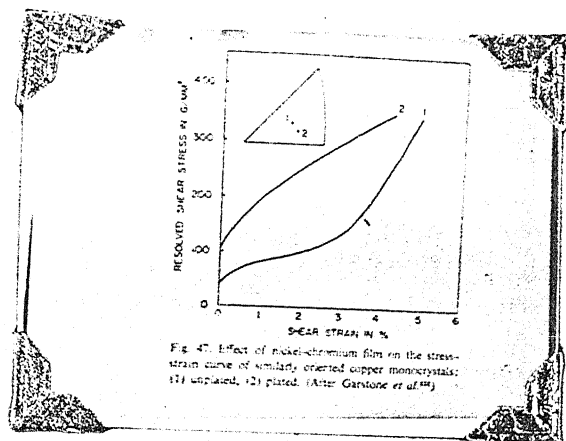


FIG. 9

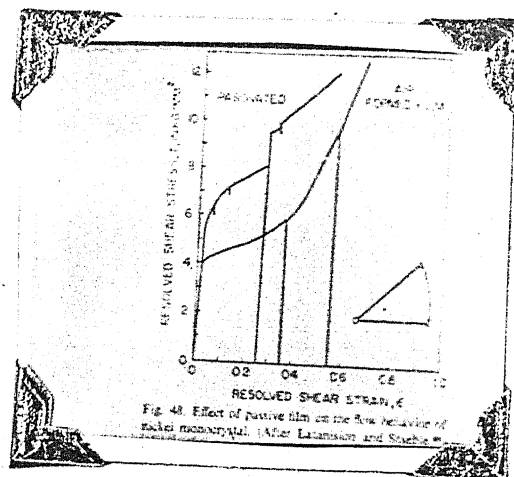


FIG. 10

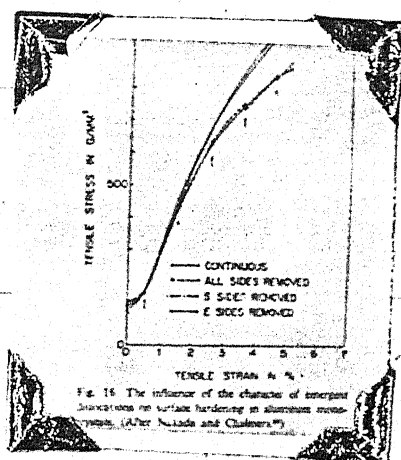


FIG. 12

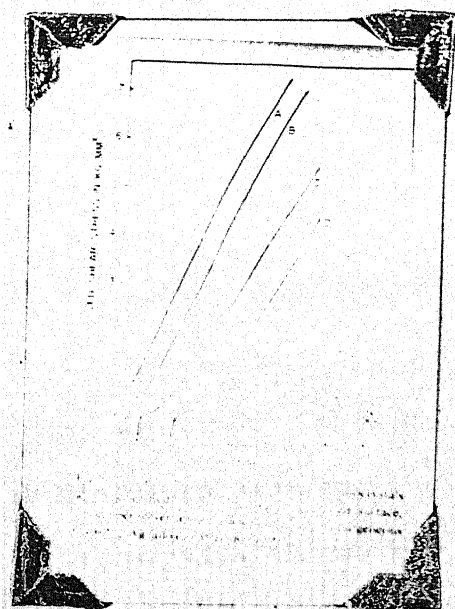


FIG. 11



FIG. 13

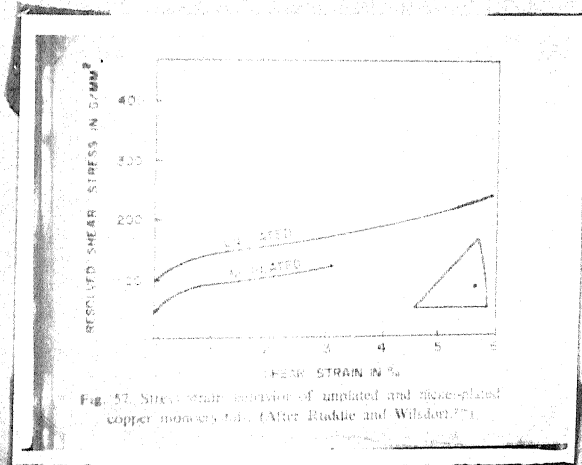


FIG. 14

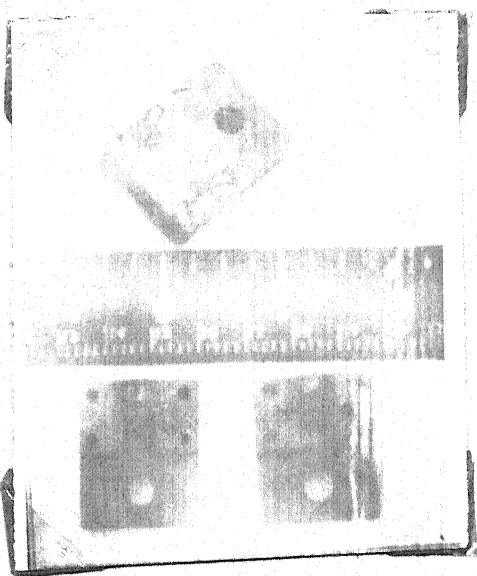


FIG. 17a BRASS GRIPS

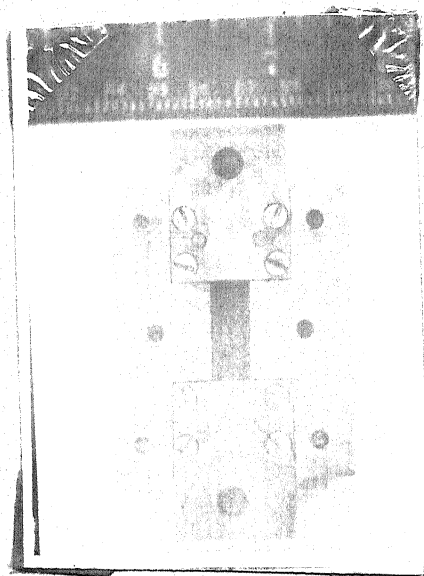


FIG. 17b GRIP ASSEMBLY

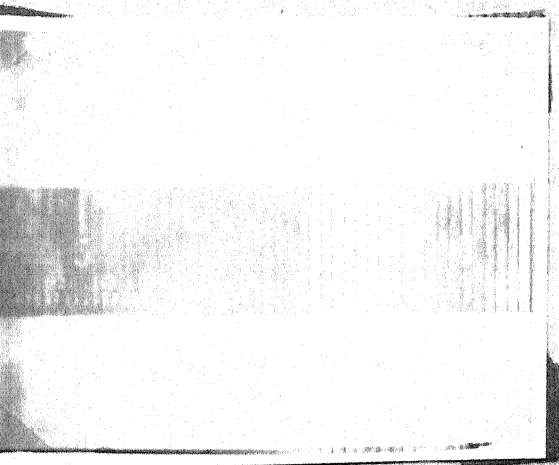


FIG. 18a TENSILE SPECIMENS

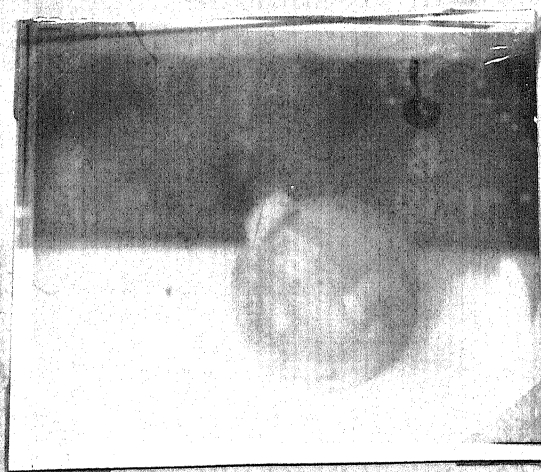


FIG. 18b SPHERICAL SINGLE CRYSTAL SEED

ME-1973-M-RAM-SUR.